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IS 4027-10 (2000): Chemical Analysis of Bronzes - Methods, Part 10: Determination of Silicon [MTD 8: Copper and Copper Alloys]



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IS 4027 (Part 10) : 2000

(Reaffirmed 2006)

भारतीय मानक

कांसे के रासायनिक विश्लेषण की पद्धतियां

भाग 10 सिलिकॉन ज्ञात करना

(पहला पुनरीक्षण)

Indian Standard

CHEMICAL ANALYSIS OF BRONZES — METHODS

PART 10 DETERMINATION OF SILICON

(First Revision)

ICS 77.120.30:71.100.55

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BUREAU OF INDIAN STANDARDS
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FOREWORD

This Indian Standard (Part 10) (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Methods of Chemical/Instrumental Analysis of Non-ferrous Metals Sectional Committee had been approved by the Metallurgical Engineering Division Council.

IS 4027 was first published in 1967 and covered determination of Cu, Pb, Sn, Mn, P, Ni, Fe, Si, AL, Zn and Sb in bronzes. While reviewing this standard the committee decided that it was convenient to revise it in series of parts which on publication will supersede the relevant method for determination given in IS 4027 : 1967 'Methods of chemical analysis of bronzes'. The other parts are as follows:

- Part 1 Determination of copper and lead by electrolytic method
- Part 2 Determination of manganese by photometric method
- Part 3 Determination of phosphorus by volumetric method
- Part 4 Determination of nickel by photometric method
- Part 5 Determination of tin by iodimetric method
- Part 6 Determination of zinc by complexometric method
- Part 7 Determination of antimony by Rhodamine B spectrophotometric method
- Part 8 Determination of iron
- Part 9 Determination of aluminium by atomic absorption spectrophotometric method
- Part 11 Determination of lead

This standard covers methods for determination of silicon in bronzes by gravimetric method (for Si > 0.1 percent) and molybdenum blue method (for Si < 0.1 percent).

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

*Indian Standard***CHEMICAL ANALYSIS OF BRONZES — METHODS****PART 10 DETERMINATION OF SILICON***(First Revision)***1 SCOPE**

This standard covers method for determination of silicon in bronzes by gravimetric method (for Si > 0.1 percent) and molybdenum blue method (for Si < 0.1 percent).

2 REFERENCE

The Indian Standard listed below contains provisions which through reference in this text, constitutes provisions of this standard. At the time of publication the edition indicated was valid. All standards are subject to revision and parties to agreement based on this standard are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below:

<i>IS No.</i>	<i>Title</i>
1817 : 1961	Methods for sampling of non-ferrous metals for chemical analysis

3 SAMPLING

Samples shall be drawn and prepared in accordance with IS 1817.

4 QUALITY OF REAGENTS

Unless specified otherwise analytical grade reagents and distilled water shall be employed in the test.

5 DETERMINATION OF SILICON BY THE GRAVIMETRIC METHOD (FOR SILICON > 0.1 PERCENT)**5.1 Outline of the Method**

Silica obtained after dehydration of the solution in the sulfuric acid medium is determined by hydrofluorization of residue as usual.

5.2 Reagents

5.2.1 Concentrated Hydrochloric Acid — rd = 1.16.

5.2.2 Concentrated Nitric Acid — rd = 1.42.

5.2.3 Concentrated Sulfuric Acid — rd = 1.84.

5.2.4 Ammonium Chloride — Solid.

5.2.5 Dilute Hydrochloric Acid — 1:5 and 1:99 (v/v).

5.2.6 Dilute Sulfuric Acid — 1:1 (v/v).

5.2.7 Hydrofluoric Acid — 40 percent.

5.3 Procedure

5.3.1 For samples containing silicon above 0.1 percent, weigh 5.00 g of sample to a porcelain vessel and dissolve in 10 ml of concentrated hydrochloric acid and 20 ml of concentrated nitric acid. When dissolution is complete add 30 ml of concentrated sulfuric acid. For samples containing 0.5 to 5 percent silicon transfer 1.00 g sample to a porcelain vessel and dissolve in 5 ml of concentrated hydrochloric acid and 10 ml of concentrated nitric acid. When solution is complete add 15 ml of concentrated sulfuric acid.

5.3.2 Evaporate on a hot plate until dense white fumes are given off. Allow to cool, add carefully 100 ml of dilute hydrochloric acid (1:5) and bring to boil. If lead is present in the alloy, add five grams of ammonium chloride and continue to boil till lead sulfate is in solution. Filter the solution immediately through close texture filter paper. Transfer completely the residue to the paper and wash thoroughly with dilute hydrochloric acid (1:99). Reserve the residue.

5.3.3 Return the filtrate to the original vessel and treat in accordance with 4.3.2. Transfer both the papers and residues to a platinum crucible. Ignite at a low temperature until the paper has been burnt off and then heat up to 1 050°C. Cool in a desiccator and weigh. Add one or two drops of dilute sulfuric acid and sufficient hydrofluoric acid (2 to 5 ml) to dissolve the residue and evaporate the solution slowly to dense white fumes. Continue to heat the crucible until all the free sulfur trioxide has been expelled and then ignite at 1 050°C for 5 min. Cool in a desiccator and weigh.

5.3.4 Make a blank determination following the same procedure and using the same amounts of all reagents but without the sample.

5.4 Calculations

$$\text{Silicon percent} = \frac{A - B}{C} \times 46.72$$

where

A = mass in g of silica obtained after HF treatment ($X_1 - X_2$),

B = mass in g of silica obtained in blank after HF treatment,

C = mass in g of sample taken,

X_1 = mass of residue before HF treatment, and

X_2 = mass of residue after HF treatment.

6 DETERMINATION OF SILICON BY MOLYBDENUM BLUE SPECTROPHOTOMETRIC METHOD (FOR SILICON < 0.1 PERCENT)

6.1 Outline of the Method

A suitable aliquot of the test solution is treated with ammonium molybdate to form molybdosilicate (yellow) complex. Complex is reduced to molybdenum blue complex with ascorbic acid and measured at 810 nm.

6.2 Reagents

6.2.1 Concentrated Hydrochloric Acid — rd = 1.16.

6.2.2 Concentrated Nitric Acid — rd = 1.42.

6.2.3 Concentrated Sulfuric Acid — rd = 1.84.

6.2.4 Dilute Hydrochloric Acid — 1:5 and 1:99 (v/v).

6.2.5 Sodium Peroxide

6.2.6 Ammonium Molybdate — 10 percent.

6.2.7 Oxalic Acid — 5 percent.

6.2.8 Ascorbic Acid — 4 percent.

6.2.9 Standard Silicon Solution (1 ml = 1 mg Si)

Weigh 2.139 3 g calcined high purity silica into a platinum crucible. Mix with 16 g of sodium carbonate and fuse at 1 050°C for 30 min. Extract the fused mass with water and transfer to 1 litre volumetric flask, dilute to mark and transfer to PTFA bottle. Transfer 10 ml of the above solution and dilute to 500 ml. Transfer to a PTFA bottle. 1 ml = 20 µg Si.

7 PROCEDURE

7.1 Weigh 0.500 g of sample in a porcelain vessel, dissolve in 5 ml concentrated hydrochloric acid and 10 ml concentrated nitric acid. Evaporate on a hot plate until dense white fumes are given off. Allow to cool. Add 100 ml of dilute hydrochloric acid (1:5) and boil. Cool filter through close texture filter paper and wash the residue with dilute hydrochloric acid (1:99). Reserve the residue and filtrate.

7.2 Transfer residue and filter paper in a platinum crucible and ignite at low temperature at 600°C. Cool and mix the residue with 0.25 g sodium peroxide and heat in a furnace at 600°C for 10 min. Cool, add 15 ml water. Cover the crucible with lid and allow the reaction to subside. Add 15 ml concentrated sulfuric acid and transfer the filtrate obtained above (7.1) and the contents of the platinum crucible to one litre volumetric flask. Dilute to mark and mix. Transfer immediately to a polypropylene or polytetrafluoroethylene container.

7.3 Pipette two 20 ml aliquots from the test solution into separate 50 ml volumetric flasks. In each case one aliquot is for test and another is for compensating solution. Add the reagents in the following manner:

a) *Test Solution* — Add in the following order shaking after each additions:

— 10 ml of ammonium molybdate and allow to stand for 20 min

— 5 ml of dilute sulfuric acid (1:4)

— 5 ml of oxalic acid and

— 5 ml of ascorbic acid solution

b) *Compensating Solution* — Add in the following order shaking after each addition:

— 5 ml of dilute sulfuric acid (1:4)

— 5 ml of oxalic acid and immediately add

— 5 ml of ascorbic acid

Dilute to mark and mix. Allow each solution to stand for 30 min.

7.4 Blank

Carry out a blank of reagents used above using 0.500 g of pure copper instead of test portion. Prepare a separate compensating solution for the blank.

7.5 Calibration Curve

Transfer 0.5 g of pure copper into 250 ml PTFA beaker and dissolve as in case of test portion and make up to 1 litre. Take 20 ml of aliquot of this solution into seven volumetric flasks and add 0, 2.5, 5, 7.5, 10, 20 and 25 ml of standard silicon solution of 20 µmg/ml (see 5.2.9) and develop the colour as mentioned above omitting the compensating and blank solution.

NOTE — Blank and compensating solutions are not necessary for the calibration tests as both are corrected via the zero aliquot of the series.

Measure the absorbance value at 810 nm using a suitable path length cell. Prepare the calibration

curve by plotting net absorbance values against the silicon concentrations expressed in µg/ml in the measured solutions.

7.6 Calculation

Convert the corrected absorbance of each test solution (test and blanks) into corresponding concentration of silicon in µg/ml by using the calibration graph. Calculate the silicon content as follows:

$$\text{Silicon, percent} = \frac{(A - B)}{C} \times \frac{1}{10}$$

where

- A = mass in mg of silicon in test solution (corrected for its compensating solution),
- B = mass in mg of silicon in blank solution (corrected for its compensating solution), and
- C = mass in g of sample represented by aliquot.

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